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James L. Smialek, Charles A. Barrett, and Ralph G. Garlick
Glenn Research Center, Cleveland, Ohio

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Acknowledgments

Helpful discussions with Dr. Rebecca MacKay, Dr. Robert Dreshfield, and Dr. John Schaeffer are greatly appreciated.

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THE EFFECT OF HYDROGEN ANNEALING ON THE OXIDATION RESISTANCE OF FOUR EPM SINGLE CRYSTAL SUPERALLOYS

James L. Smialek, Charles A. Barrett, and Ralph G. Garlick
National Aeronautics and Space Administration
Glenn Research Center
Cleveland, Ohio 44135

SUMMARY

Four single crystal EPM developmental airfoil superalloys were hydrogen annealed at 1300 °C for up to 100 hr to remove sulfur and improve oxidation resistance. Although the 1100 and 1150 °C cyclic oxidation resistance was remarkably improved by annealing for 24 or 100 hr, the behavior was still considerably inferior to that of commercially available single crystal superalloys, especially those that are either Y-doped or hydrogen annealed. Excessive degradation in the developmental alloys appeared to be correlated with low Cr contents and, to a lesser extent, high Co and Re contents.

INTRODUCTION

The oxidation resistance of single crystal superalloys has improved greatly over the last decade. The addition of high levels of tantalum appears to be associated with slow growing mixed scales of CrTaO_4 and NiTa_2O_6 with the more commonly observed Al_2O_3 and $\text{Ni}(\text{Al,Cr})_2\text{O}_4$ phases formed on oxidation resistant NiCrAl alloys. Furthermore, the addition of Hf (PWA 1484, Rene'N5, and CMSX 4) and Y (PWA 1487 and Rene'N5) produce even greater oxidation resistance by the well known reactive element effect. The role of bulk sulfur content and sulfur segregation in Al_2O_3 scale adhesion has also been the topic of many studies. In the past ten years, a number of hydrogen annealing studies have shown that desulfurization of superalloys from 10 ppmw S down to 0.1 ppmw S is readily possible and that the resultant oxidation resistance is comparable to Y-doped alloys, many of which are covered in a recent review article (ref. 1).

Since the EPM turbine airfoil alloy development program was primarily directed toward improved high temperature mechanical properties, Y additions were postponed to a later date, and scale adhesion issues for the uncoated alloys were not specifically addressed. The purpose of this study is to provide baseline cyclic oxidation resistance of early EPM alloys and to demonstrate what level of improvements are possible through desulfurization by hydrogen annealing. A concurrent objective is to determine what annealing schedule is necessary for sufficient desulfurization. The response of desulfurized alloys in cyclic oxidation tests should be indicative of the level of improvements that can be achieved with Y additions.

MATERIALS AND EXPERIMENTAL PROCEDURES

EPM alloys #29, 35, 39, and 41 were obtained from excess slab material cast at EMTL, GE Aircraft Engines. The nominal (target) compositions are shown in table I (ref. 2). The slab material was EDM machined into 1-mm thick coupons with surface dimensions of about 1/4 by 1/2 to 1 in. (0.6 by 1.2 to 2.0 cm) and polished to a 600 grit finish. Duplicate samples were annealed in a 5 percent H_2/Ar atmosphere (<1 ppm H_2O) at 1300 °C for 4, 24, and 100 hr.

All of the duplicate samples were tested in cyclic oxidation using 1 hr at temperature and 20 min cooling to about 60 °C. Duplicate samples of each alloy were oxidized in the as-received condition, (with a 600 grit finish), and after annealing for 4, 24, and 100 hr. The annealed samples were tested in the as-annealed condition, except for alloy 29; here one of the duplicate annealed samples was repolished to a 600 grit finish before oxidation, removing an

average of 7.2 mg/cm^2 or an $8 \text{ }\mu\text{m}$ (1/3 mil) layer, assuming a density of about 9 gm/cm^3 . Samples of alloys #29 and #35 were cyclic furnace oxidation tested at $1150 \text{ }^\circ\text{C}$ for 80 to 100 hr, when severe weight loss necessitated termination of the test. Alloys #39 and #41 were oxidized at either $1150 \text{ }^\circ\text{C}$ for 100 hr or at $1100 \text{ }^\circ\text{C}$ for 200 hr. Weight changes were recorded throughout the test. The oxide scale phases formed on the oxidized coupons were analyzed by X-ray diffraction.

RESULTS

Hydrogen Annealing

The annealing process produced average weight changes of only $+0.001$, and -0.010 mg/cm^2 , respectively, for the three annealing times. All samples were extremely reflective and metallic in appearance after annealing, indicating that no oxide scale formed during annealing. Therefore, no barrier layer existed and desulfurization should have proceeded as expected.

Cyclic Oxidation Weight Change

The specific weight change data for $1150 \text{ }^\circ\text{C}$ cyclic oxidation of alloy #29 is shown in figure 1. Rather severe weight losses were exhibited for all the samples. No differences were noted between samples tested with the as-annealed surface condition and those re-polished to a 600 grit surface finish. Hydrogen annealing at $1300 \text{ }^\circ\text{C}$ for 24 to 100 hr did reduce the weight loss somewhat from 180 to 190 mg/cm^2 down to 110 to 135 mg/cm^2 . However, essentially all the annealing times were ineffective in producing a truly protective scale at $1150 \text{ }^\circ\text{C}$, despite some improvement. In comparison, second generation SX superalloys, Rene'N5 and PWA 1484, with either Y-doping or hydrogen annealing, gain $<1 \text{ mg/cm}^2$ after 1000 1-hr cycles to $1150 \text{ }^\circ\text{C}$ (refs. 3 to 5).

The weight change data for $1150 \text{ }^\circ\text{C}$ cyclic oxidation of alloy #35 is shown in figure 2. Again large weight losses (100 mg/cm^2) were exhibited by the as-received samples test for only 100 hr at $1150 \text{ }^\circ\text{C}$, but these were somewhat less severe than those obtained for alloy #29. Furthermore, a more significant effect of 24 and 100 hr hydrogen annealing occurred for alloy #35, decreasing the weight loss to only 15 to 18 mg/cm^2 after 100 hr of oxidation. Identical behavior was displayed by the duplicate samples, both tested in the as-annealed surface condition.

The $1150 \text{ }^\circ\text{C}$ data for alloys #39 and #41 are shown in figures 3 and 4, respectively. Both alloys show similar performance, falling between those of the previously discussed alloys. The duplicate samples of these alloys were not tested as pairs. Rather, because of the poor overall performance and absence of any surface condition effects, these duplicate samples were tested at a less severe temperature to produce more meaningful results. Accordingly, the results of $1100 \text{ }^\circ\text{C}$ cyclic oxidation testing is shown in figures 5 and 6. Although both as-received alloys still exhibited substantial weight loss ($>150 \text{ mg/cm}^2$), alloy #39, when hydrogen annealed for 24 or 100 hr, achieved a weight loss of only 6 mg/cm^2 after 200 hr at $1100 \text{ }^\circ\text{C}$.

The comparative behavior of the four alloys can be readily assessed in figures 7 and 8, where the final weight change from the 1150 and $1100 \text{ }^\circ\text{C}$ tests (for as-annealed surfaces) are plotted as a function of hydrogen annealing time. A number of points are clear from this figure: (1) hydrogen annealing at $1300 \text{ }^\circ\text{C}$ produced substantial improvements for all the alloys; (2) alloys #35 and #39 are superior to alloys #41 and #29 after hydrogen annealing; and (3) hydrogen annealing for 24 hr produced most of the benefit, with little further improvement demonstrated for 100 hr annealing.

X-ray Diffraction

No initial protective scales were preferentially formed from the hydrogen annealing process. The scale phases present at the end of the oxidation test were characterized by X-ray diffraction on a select number of specimens. The results are summarized in table II. The major scale phases were NiO, NiTa_2O_6 , and $\text{Ni(Al,Cr)}_2\text{O}_4$ spinel. Very little Al_2O_3 was identified, primarily because of the masking effect of the other heavily absorbing oxide phases and the severity of attack sustained at the end of testing. Thus, the protective nature of the scale was limited. Surprisingly,

no trend in scale type was observed as a function of performance (annealing time or alloy composition). It should be noted that the lattice parameter of NiAl_2O_4 is 8.05\AA , and that for NiCr_2O_4 is 8.30\AA . Thus the observed spinel represents a mixed composition somewhat closer to nickel aluminate than to nickel chromate.

Alloy Composition Effects

The major compositional variations in this set of four alloys occur for Cr, Co, Ru, and Re (table I). These are also the elements that have substantial differences in level from those in the two commercial alloys listed. The alloys are also ranked according to the final weight changes. This ranking remained relatively consistent for all the hydrogen pre-anneals, as well as for the as received material, figure 7. (The weight change data in table I is that for 100 hr oxidation at 1150°C , for EPM samples having 100 hr hydrogen annealing at 1300°C , and for the commercial alloys annealed at 1250°C for 50 to 100 hr.). There is only one compositional change in going from the best to second best alloy (#35 to #39, i.e., an increase in Co from 5 to 20 percent. In surveying an extensive database for Ni(Co) superalloys, there is rarely any correlation with performance and Co content, (ref. 6).

A more significant performance loss is incurred in going from alloys #39 to #41 by reducing Ru from 4.5 to 1.5 percent. And finally, the worst performance of alloy #29 is obtained by reducing Cr from 2.3 to 1.5 percent, decreasing Ru from 1.5 to 1.0 percent, and increasing Re from 5.7 to 6.25 percent. There is some reasonable precedent to suggest that low Cr can be deleterious, in that some Cr is needed to assist in a transient oxidation period during which a protective Al_2O_3 scale is being built up. Cr has been weakly correlated with improved cyclic oxidation resistance in superalloys overall, but generally at levels higher than 5 percent (ref. 6). Previous EPM experience (Dr. Jon C. Schaeffer) would indicate that Cr and Ru may act in concert to produce beneficial effects.

However these performance changes must be evaluated in the context of the excellent performance of current oxidation resistant single crystal alloys, containing 5 to 7 percent Cr, 8 to 10 percent Cr, 3 percent Re, 5.6 to 6.2 percent Al, and no Ru. For example, weight loss of only 5 to 7 mg/cm^2 were achieved for as-received Rene'N5 and PWA 1484 (without Y) after 100 hr of cyclic oxidation at 1150°C or with weight gains of only 0.6 to 2 mg/cm^2 after 1000 hr for hydrogen annealed Rene'N5 and PWA 1484 (refs. 3 to 5). Similar results were obtained for 1100 to 1200°C tests of hydrogen annealed PWA 1480 and CMSX-4 (refs. 7 to 9).

These alloys possess substantial compositional differences with the four EPM alloys, as presented in table I. The most critical difference is believed to be the Cr content, which is only 1.5 to 2.3 percent for the EPM alloys compared to 5 to 6 percent in current single crystal superalloys. Aluminum content is always very important, and the EPM alloys do contain 5.5 percent Al. The Co levels at least overlap current alloys, but 20 percent Co appears to be relatively high. The Re levels did not vary appreciably in the four alloy set, but are considerably higher than present alloys, which contain 3 percent. Finally, higher Ru levels were identified in the best EPM alloys, but no Ru is present in the superior conventional single crystal alloys. Thus Ru may help in the EPM alloys, but is not needed for the good oxidation resistance of current superalloys.

In summary, low Cr levels are believed to have had the most detrimental effect on oxidation resistance. Also, high Re may have degraded performance.

Sulfur Content

The sulfur contents were not measured, but the trend with hydrogen annealing could be estimated from the thin slab diffusion equation, as demonstrated previously (ref. 3). Assuming sulfur diffusion control of desulfurization, the thin slab diffusion solution provides the following approximation (for values of $Dt/L^2 > 0.05$):

$$C_s/C_o = 8/\pi^2 \exp(-\pi^2 D_s t/L^2) \quad (1)$$

where C_s and C_o are the final and original average sulfur contents ($<<1$ percent), D_s is the diffusion coefficient of sulfur in nickel, t is time, and L is thickness. The results of the two best oxidation runs (#35 at 1150°C and #39 at 1100°C) are shown in figure 9 as a function of the estimated sulfur content, as determined for the various annealing times. Here the oxidation response can be seen to steadily improve as the estimated sulfur content falls below

1 ppmw, assuming a starting level of 6.3 ppmw. (This sulfur level was taken from a previous study of Rene'142 (ref. 3), more recent studies show sulfur levels of 2.6 ppmw for Rene'N5 [5].)

(It should be noted that the 100 hr anneal would not actually produce the calculated level of 10^{-12} ppm sulfur. Hydrogen annealing has been known to produce a minimum as low as 0.01 ppmw sulfur, but usually about 0.1 ppmw, both of which are nonetheless very low. In any event, the conclusion reached from this type of plot is that the major effect of desulfurizing a 1-mm thick coupon, as determined in cyclic oxidation tests, occurs at durations between 4 and 24 hr of hydrogen annealing.)

The plot is consistent with published summaries of hydrogen annealed superalloys which showed diminishing returns below about 0.1 to 0.3 ppmw. This is also about the level that is equivalent to one monolayer of sulfur, if all the bulk impurity were to segregate to the interface (refs. 3, 4, and 10). Finally, it is very similar in form to plots of weight change versus calculated and actual sulfur content for PWA 1480 (refs. 4 and 11). But in these studies the 1100 °C oxidation resistance was much better overall, such that the final weights attained after 1000 1-hr cycles were +0.5 to +1 mg/cm².

CONCLUSIONS

This screening study has shown that some representative EPM single crystal alloys presently possess significant debits compared to the oxidation resistance of commercially available superalloys. It is believed that the single most important cause of this debit is the low Cr content, with lesser contributions possible from the high Co and Re contents. Nonetheless, hydrogen annealing (desulfurizing) did have a strong positive effect, suggesting that low sulfur alloys and Y additions would provide some useful benefits to the oxidation behavior of this class of alloys. However these modifications would not produce oxidation resistance comparable to that of current second generation single crystal superalloys.

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TABLE I.—NOMINAL CHEMISTRIES (wt %) OF FOUR SAMPLE EPM DEVELOPMENTAL SINGLE CRYSTAL AIRFOIL SUPERALLOYS AND TWO COMMERCIAL ALLOYS. ORDERED ACCORDING TO WEIGHT CHANGE AFTER 100 1-hr CYCLES AT 1150 °C FOR EPM SAMPLES THAT WERE HYDROGEN ANNEALED AT 1300 °C FOR 100 hr, AND FOR COMMERCIAL ALLOYS HYDROGEN ANNEALED AT 1250 °C FOR 50 TO 100 hr

Commercial alloy	Al	Ta	Cr	Co	Ru	Re	W	Mo	Ti	$\Delta W/A_{1150\text{ }^{\circ}\text{C}, 100\text{ hr}}$ mg/cm ²
Rene'N5	6.2	7	7	8	0	3	5	2	0	0.6
PWA 1484	5.6	9	5	10	0	3	6	2	0	0.6
EPM alloy										
#35	5.5	8.3	2.3	5.0	4.5	5.7	5.6	2.0	0.4	-14.9
#39	5.5	8.3	2.3	20.0	4.5	5.7	5.6	2.0	0.4	-22.7
#41	5.5	8.3	2.3	20.0	1.5	5.7	5.6	2.0	0.4	-64.5
#29	5.6	8.0	1.5	20.0	1.0	6.2	6.0	2.0	0.0	^a (-160)

^aExtrapolated.

TABLE II.—SUMMARY OF SCALE PHASES FORMED AFTER CYCLIC OXIDATION

	Alloy #35 600 grit	Alloy #39 as-annealed	Alloy #41 as-annealed
	1150 °C, 100 hr	1150 °C, 100 hr	1150 °C, 100 hr
1300 °C anneal time			
0 hr	N(p), N-T, S(8.10)	N-T, N, S(8.10)	N, N-T, S(8.10)
4 hr	N(p), N-T, S(8.10)	N-T, N, S(8.10)	N-T, N, S(8.10)
24 hr	N(p), N-T, S(8.10)	N, N-T, S(8.10)	N-T, N, S(8.10)
100 hr	N(p), N-T, S(8.10), A	N, N-T, S(8.10)	N-T, N, S(8.10)
		1100 °C, 200 hr	1100 °C, 200 hr
1300 °C anneal time			
0 hr		N(p), S(8.20), N-T	N, N-T, S(8.10)
4 hr		N(p), S(8.15), N-T	N, N-T, S(8.10)
24 hr		N(p), S(8.20), N-T	N, N-T, S(8.10)
100 hr		N(p), S(8.20), N-T	N, N-T, S(8.10)

N = NiO

N-T = NiTa₂O₆

S = Ni(Al,Cr)₂O₄

A = Al₂O₃

(p) = preferred orientation

(8.10) = a₀

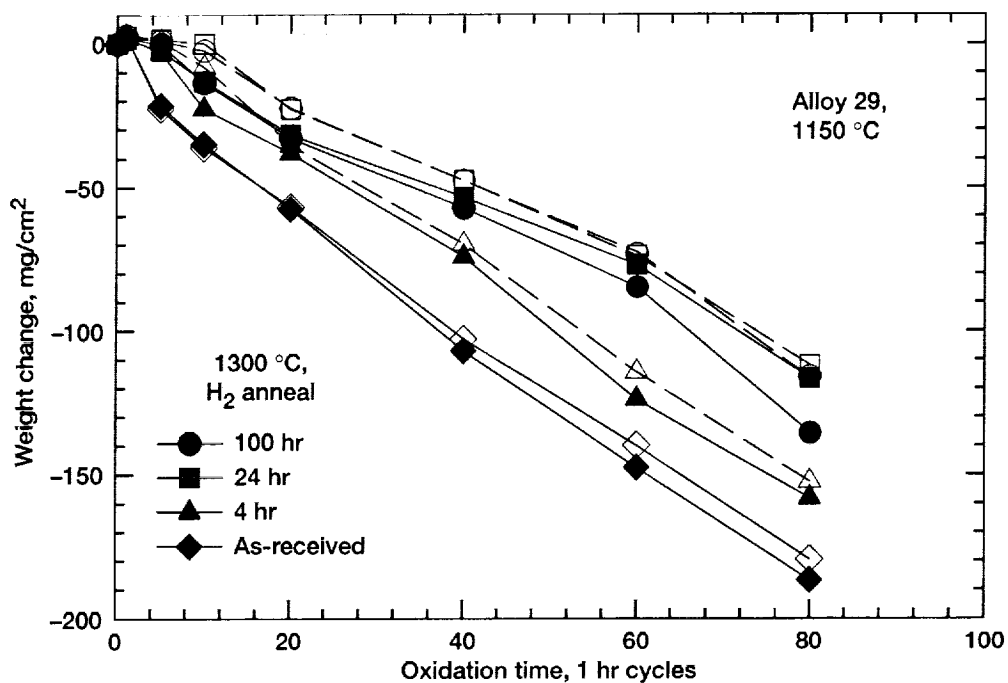


Figure 1.—Effect of hydrogen annealing times on the oxidation resistance of alloy #29. (1150 °C, 1 hr cycles). As-annealed surface condition (dashed lines) shows little difference with 600 grit polished surfaces (solid lines).

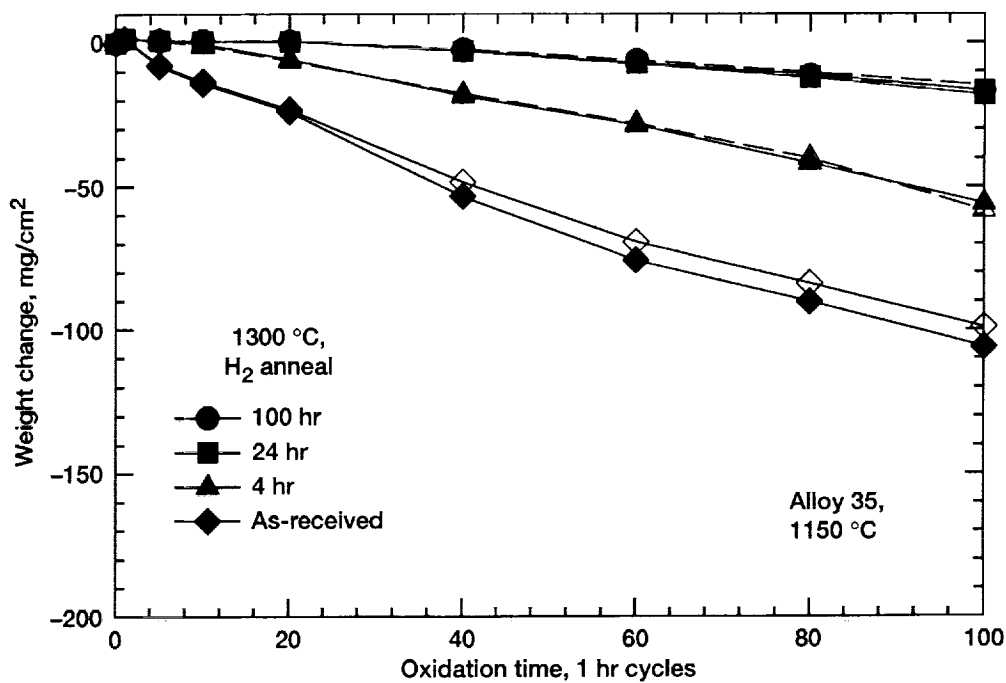


Figure 2.—Effect of hydrogen annealing times on the oxidation resistance of alloy #35. (1150 °C, 1 hr cycles). As-annealed surface (dashed lines); similar behavior of duplicate samples.

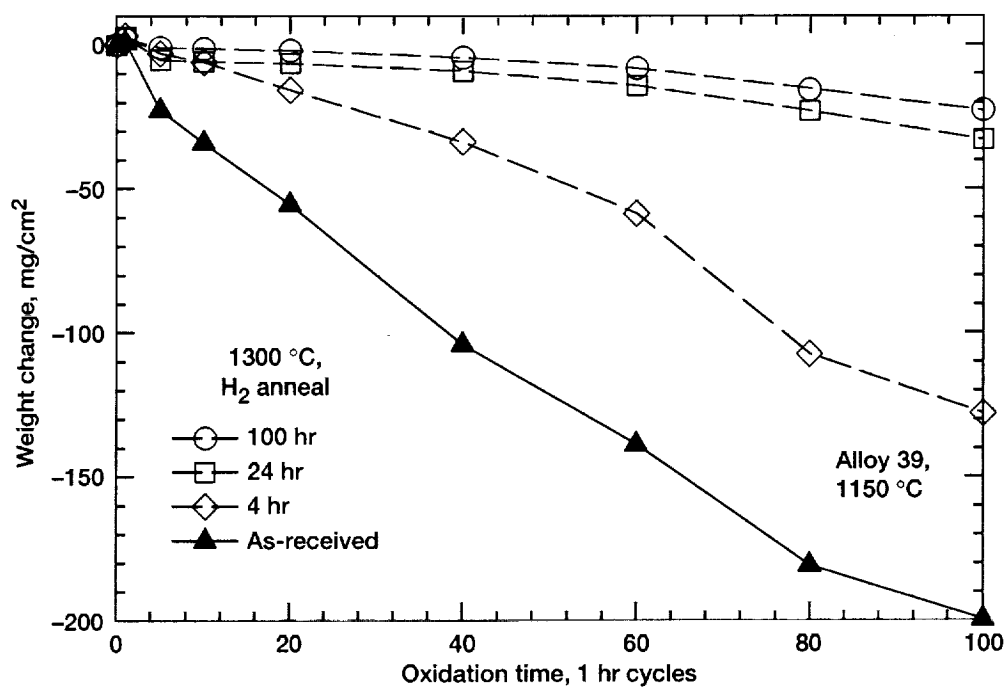


Figure 3.—Effect of hydrogen annealing times on the oxidation resistance of alloy #39. (1150 °C, 1 hr cycles; dashed lines: as-annealed surfaces).

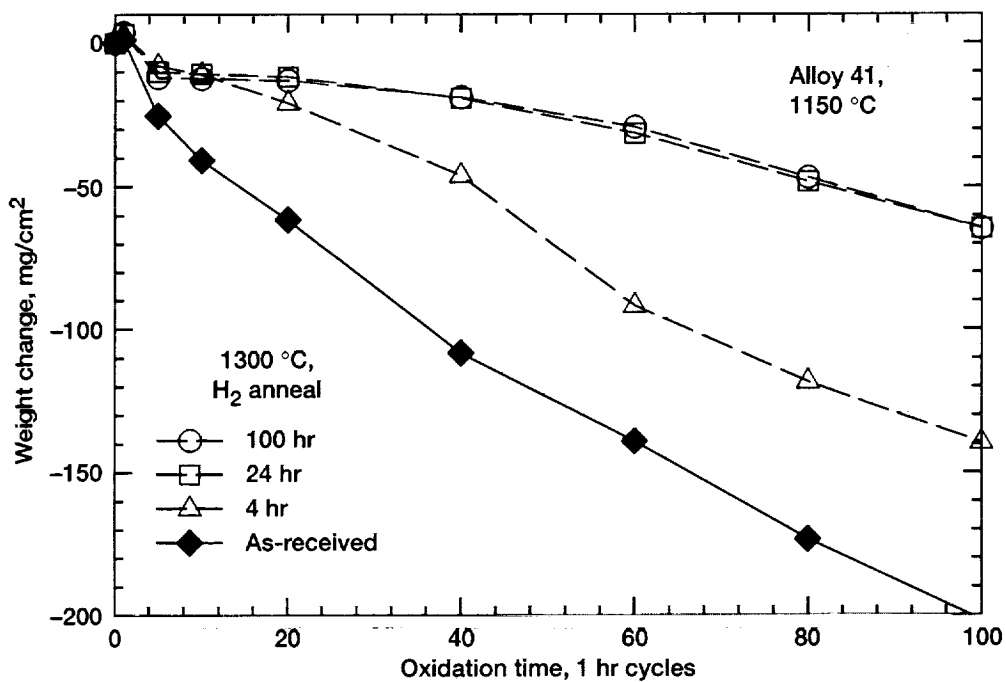


Figure 4.—Effect of hydrogen annealing times on the oxidation resistance of alloy #41. (1150 °C, 1 hr cycles; dashed lines: as-annealed surfaces).

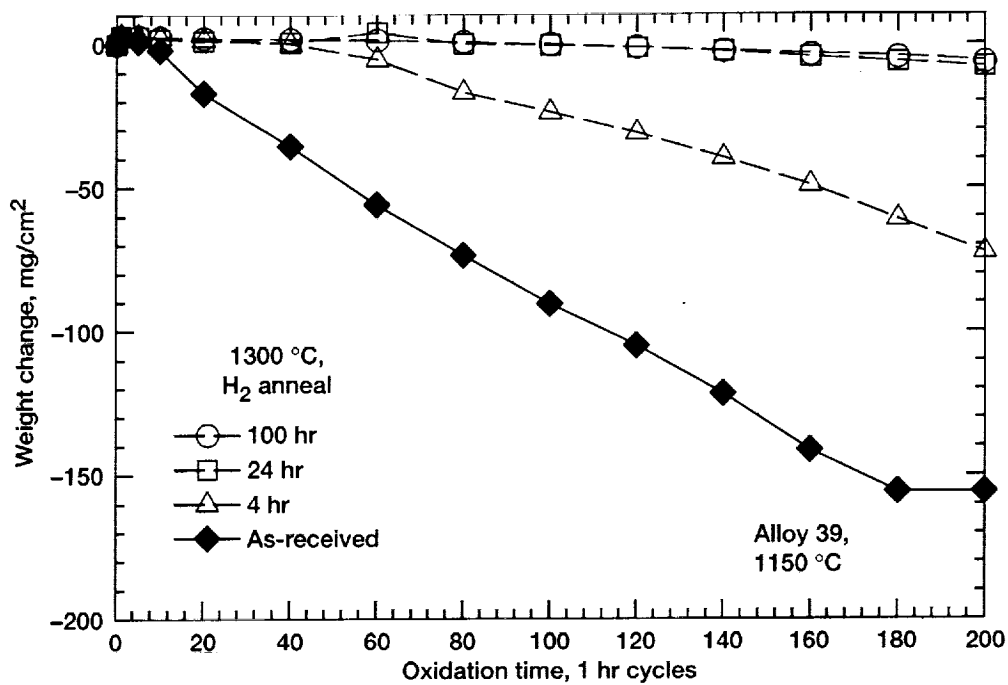


Figure 5.—Effect of hydrogen annealing times on the oxidation resistance of alloy #39. (1100 °C, 1 hr cycles; dashed lines: as-annealed surfaces).

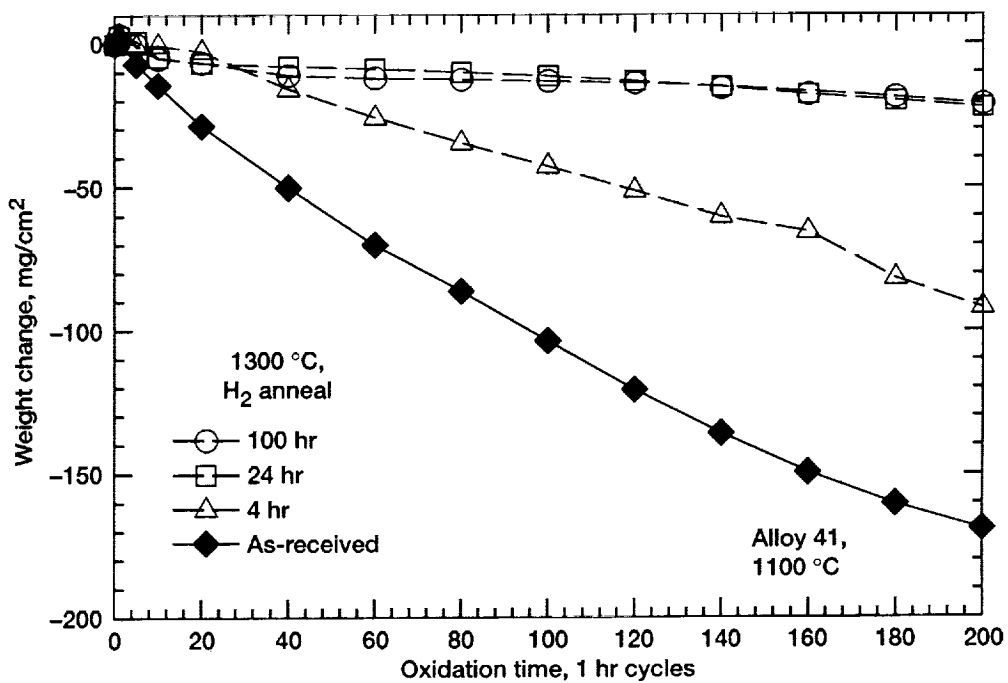


Figure 6.—Effect of hydrogen annealing times on the oxidation resistance of alloy #41. (1100 °C, 1 hr cycles; dashed lines: as-annealed surfaces).

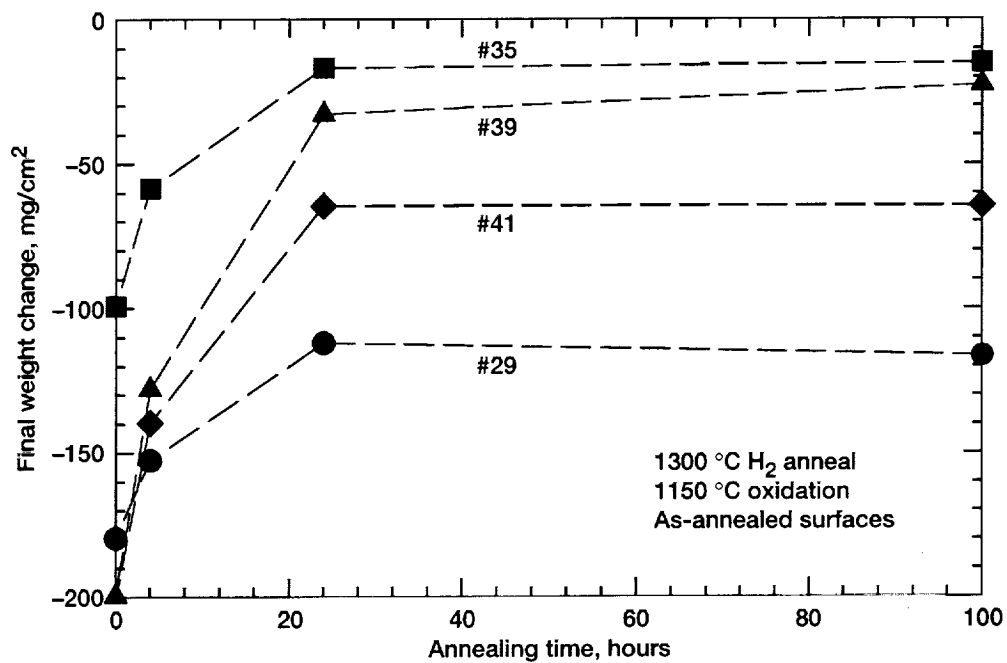


Figure 7.—Effect of alloy type and hydrogen annealing times on the final weight change of EPM alloys. (1150 °C, 1 hr cycle oxidation; 1300 °C annealing, as-annealed surfaces).

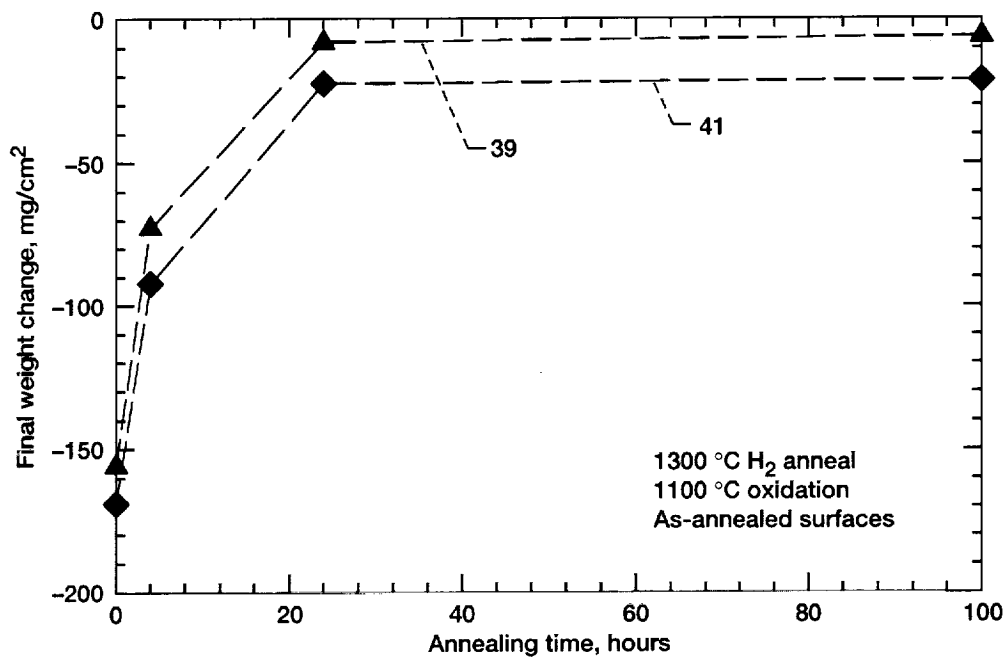


Figure 8.—Effect of alloy type and hydrogen annealing times on the final weight change of EPM alloys. (1100 °C, 1 hr cycle oxidation; 1300 °C annealing, as-annealed surfaces).

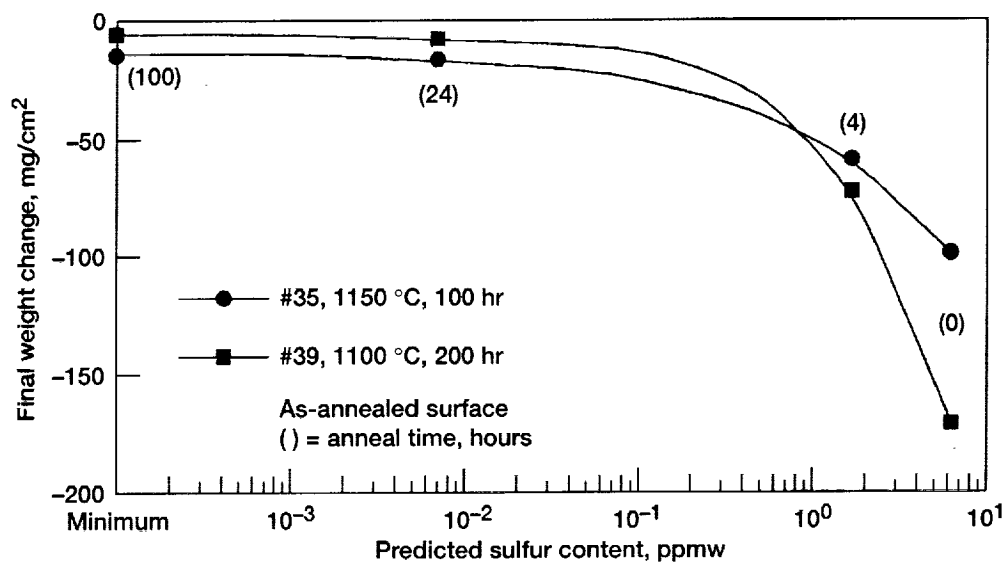


Figure 9.—Correlation of final weight change with the predicted sulfur content of hydrogen annealed EPM alloys. (1300 °C anneal, as-annealed surfaces, 6.3 ppmw initial sulfur assumed).

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1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE August 2001		3. REPORT TYPE AND DATES COVERED Technical Memorandum
4. TITLE AND SUBTITLE The Effect of Hydrogen Annealing on the Oxidation Resistance of Four EPM Single Crystal Superalloys			5. FUNDING NUMBERS WU-714-04-20-00	
6. AUTHOR(S) James L. Smialek, Charles A. Barrett, and Ralph G. Garlick				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) National Aeronautics and Space Administration John H. Glenn Research Center at Lewis Field Cleveland, Ohio 44135-3191			8. PERFORMING ORGANIZATION REPORT NUMBER E-12883	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) National Aeronautics and Space Administration Washington, DC 20546-0001			10. SPONSORING/MONITORING AGENCY REPORT NUMBER NASA TM-2001-211064	
11. SUPPLEMENTARY NOTES Responsible person, James L. Smialek, organization code 5160, 216-433-5500.				
12a. DISTRIBUTION/AVAILABILITY STATEMENT Unclassified - Unlimited Subject Category: 26 Available electronically at http://gltrs.grc.nasa.gov/GLTRS This publication is available from the NASA Center for Aerospace Information, 301-621-0390.			12b. DISTRIBUTION CODE	
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14. SUBJECT TERMS Metallic materials			15. NUMBER OF PAGES 16	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT	